RATE STUDIES IN MOLECULAR DISTILLATION

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ABSTRACT

This investigation was made to study the effects of surface temperature, system pressure, and mechanical stirring on the distillation rate of a pot type molecular still. There is in the literature to date a negligable amount of data on this type of still. The material used was di-butyl pthalate. It was found that the rate increases and the efficiency decreases as the temperature increases. The "critical pressure" (highest pressure under which satisfactory molecular distillation will take place in this still) was found to be o.03 microns. The nature of the distillation was found to change at this pressure. Mechanical stirring has no appreciable effect on the rate. The results obtained are in substantial agreement with the few results obtained by previous workers in this field even though different types of stills and different materials were used.

NOMENCLATURE

A - area of evaporation surface

D - distillability

d - molecular diameter

k - proportionality constant

L - mean free path

M - molecular weight

m - evaporation rate ($gm/sec/cm^2$)

N - concentration

n - evaporation rate (gm/sec)

P - total pressure

p - partial pressure or vapor pressure

Q - fraction of material distilled

R - gas constant

S - time

T - temperature (^oKelvin)

T_B - normal boiling point

t - temperature ($^oCentigrade$)

v - distillation rate (elimination)

W - distillation rate (actual)

ϕ - Washburn's constant

subscripts

c - condenser condition

e - evaporator condition

i - time interval number

RATE STUDIES IN MOLECULAR DISTILLATION

INTRODUCTION

Molecular distillation is defined as the process of distillation from a heated surface to a nearby cooled surface with the intervening space highly evacuated so that the mean free path of the distilling molecules is equal to or greater than the distance from the evaporating surface to the condensing surface.[1]

Under ordinary pressures distillation takes place only after the material to be distilled begins to boil. This boiling point is by definition the point at which the vapor pressure of the material becomes equal to the pressure of the system. In molecular distillation it is not necessary for the material to be heated to its boiling point; and consequently, distillation may take place at a lower temperature. In this respect molecular distillation must not be confused with high-vacuum distillation which requires that the material be heated to the boiling point.

Some high molecular weight compounds with very low vapor pressures, such as heavy oils and fats, which might possibly undergo thermal decomposition if distilled by ordinary means, may be distilled by molecular distillation methods.

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[1]Embree, N.D.; Chemical Reviews, 29, 317 (1941)

The pressure in the system must be reduced low enough so that the distilling molecules will pass from the evaporating surface to the condensing surface without colliding with other molecules or any obstructing surface.[2]

Under these low pressure conditions there is no sharply defined temperature at which distillation will begin. Distillation starts as soon as the pressure in the system is low enough so that the mean free path of the distilling molecules is of the same order of magnitude as the distance from the evaporator to the condenser. Once this pressure is obtained, further reduction in pressure has no effect on the distillation rate. The controlling factor then becomes the temperature of the evaporating surface. A fundamental concept applying to low pressure distillation is that the vapor pressure of the distilland is a function of temperature only.

This work is primarily concerned with making a study of the effect of temperature on the rate of distillation in a pot type still. An attempt was made to increase the rate by mechancial agitation and also to determine the critical pressure needed for molecular distillation.

The material used throughout this work is di-butyl pthalate.

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[2]Howat, D.D.; Chemical Age, 45, 309 (1941)

HISTORICAL DEVELOPMENT

Initial Development

Molecular distillation was first studied by J. N. Bronsted and G. Hevesy[3] who devised by its use a method of separating the isotopes of mercury. The apparatus used consisted of two concentric flasks sealed as shown in Figure 1. The inner flask, A, was filled with liquid air and served as

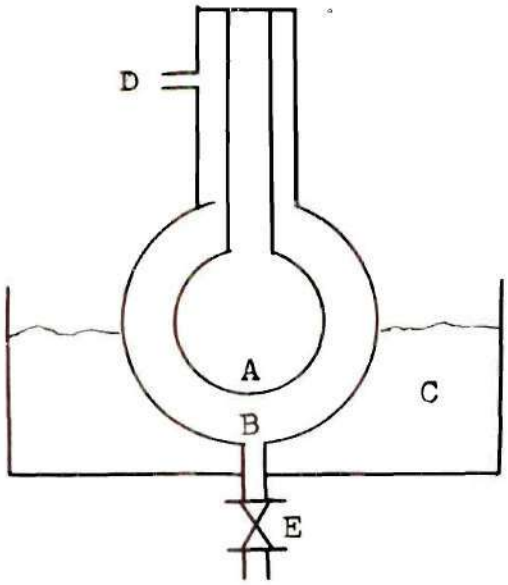

FIGURE 1: BRONSTED AND HEVESY STILL

a condenser. The outer flask, B, was partially filled with mercury and the space between the two evacuated by means of a condensation pump at D. The outer flask was then heated in an oil bath, C, and the molecules evaporating from the outer

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[3]Bronsted, J.N. and Hevesy, G.; Philosophical Magazine, 43, 31 (1922)

flask were condensed and frozen onto the inner one. Upon completion of a run the outer flask was drained by the stopcock, E, at the bottom of the still. The distillate was then removed by allowing it to melt and run out through the same stopcock.

A few years later C. R. Burch[4] adapted this method to the distillation of organic materials. In his publication

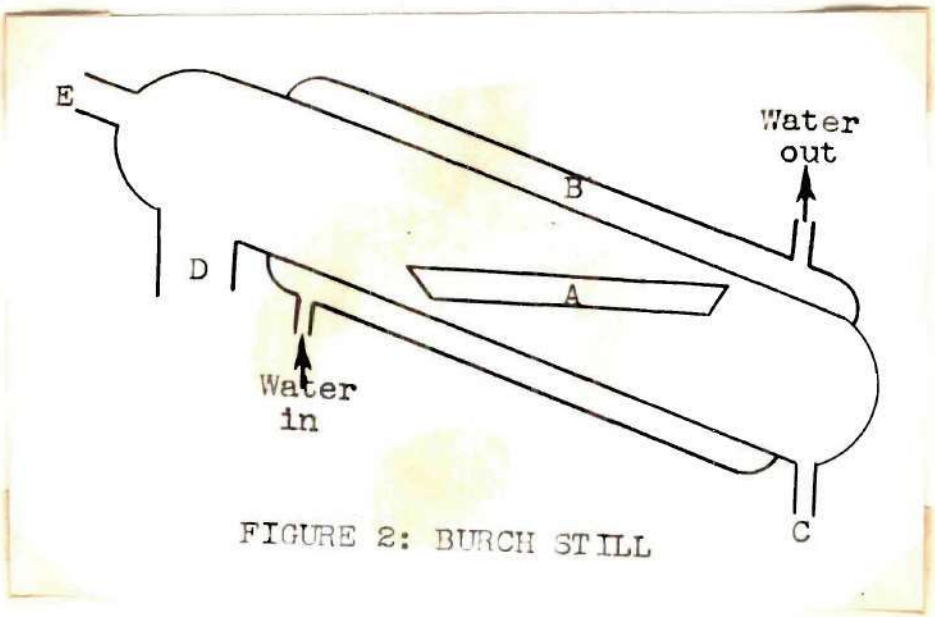

FIGURE 2: BURCH STILL

he stated that crude petroleum and petroleum derivatives had been distilled under conditions practically equivalent to evaporation into a perfect vacuum. He also found that the

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[4]Burch, C.R.; Proceedings of Royal Society (London), 123 A, 271 (1929)

high molecular weight compounds were fractionated by this molecular distillation method with no noticeable trace of decomposition. The same oils when distilled under ordinary conditions began to decompose quite readily at temperatures ranging from 307° to 340° C. Greases and oils having exceedingly low vapor pressures were found which permitted the free use of ground joints in high-vacuum systems and also the oils could replace mercury in condensation and diffusion pumps.

Burch's apparatus (Figure 2) consisted of a copper evaporating tray, A, 16 cm. long, 2.5 cm. wide, and 1.1 cm. deep, provided with an electric heater. This tray was encased in a water-jacketed glass condenser set at a slight angle to allow the condensate to flow down and out to the distillation receives at C. The high vacuum was produced by means of a condensation pump attached to the system at D. All leads and gauges were connected at E.

It was also noted that under ordinary distillation conditions bubbling or frothing accompanied the boiling of the distilland with noticeable decomposition taking place simultaneously. However, under molecular distillation conditions the distilling surface was relatively quiescent in most cases; and a separation was effected according to Langmuir's equation (see Theoretical Discussion); that is in the ratio of the square roots of the molecular weights for materials of equal vapor pressure.

At about the same time, E. W. Washburn[5] was working with molecular distillation using stills which were roughly of the same design as those used by Bronsted and Hevesy. However a slight variation was introduced which gave a greater condensation area. This design is shown in Figure 3. The evaporating surface is designated by A, the condensing surface

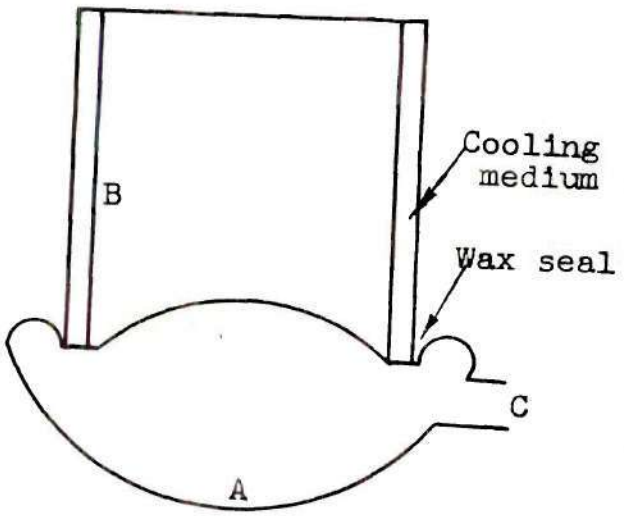

FIGURE 3: WASHBURN STILL

by B, and the opening to the high vacuum by C. The condenser is jacketed and dry ice or liquid air is used as a cooling medium. After distillation the wax seal is broken and the condenser removed.

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[5]Washburn, E.W.; National Bureau of Standards Journal of Research, 2, 476 (1929)

Like Burch, Washburn was concerned with the distillation of low vapor pressure substances which had tendencies to decompose at temperatures of ordinary vacuum distillation. In discussing the rates obtained he observed that the actual rate of distillation would always be less than the calculated value owing to some reflection of molecules from the condensing surface, but such reflection should at worse never amount to more than 90 per cent.[6] Thus the actual rate should never be less than 10 per cent of the calculated rate if the temperature of the condensing surface is low enough, if the vacuum is high enough, and if the evaporating surface is clean (i.e. free from a layer of lower volatility).

Comparison of Stills

After the early investigations the leading work in molecular distillation has been carried on by K.C.D. Hickman. Due to the great amount of work done, he is generally recognized as the foremost authority on molecular distillation. He has also contributed a great deal to the development of the various types of molecular stills.

The early stills used by Hickman were of simple construction and represent the early types of molecular stills. These stills are shown in Figure 4. In each case the evaporating surface is designated by A, the condensing surface by B,

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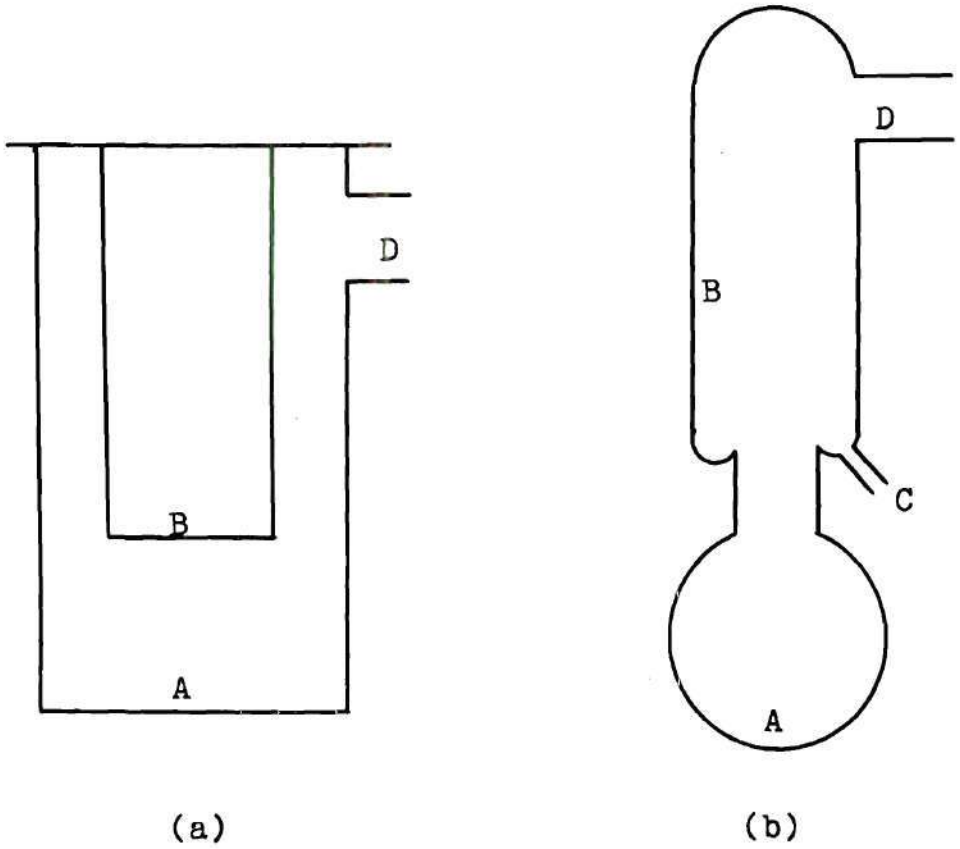
[6]Langmuir, I.; Physical Review, 7, 176 (1916)



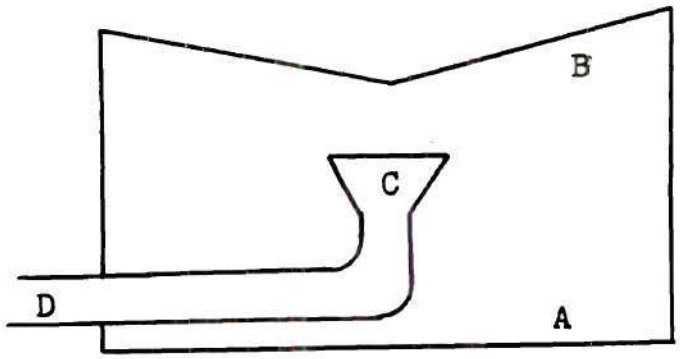


FIGURE 4: TYPES OF MOLECULAR STILLS (HICKMAN)

the distillate receiver by C, and the high-vacuum producer by D. Figures 4 (a) and 4 (b) are self-explanatory. In 4 (c) the condensate is allowed to drip into a funnel which carries the distillate to the receiver. The type still represented in 4 (a) is dependent on the condensate being frozen to the collecting surface by means of liquid air.

The main use of molecular distillation is to obtain a separation with a minimum amount of decomposition. In the pot type stills the distilland is in constant contact with the heating medium which constitutes thermal exposure and gives a possibility of decomposition. It has been found that even though decomposition of the substances being distilled is greatly reduced by molecular distillation it is not completely eliminated. Another drawback of the pot type still is that no stirring is obtained and a poor separation results, since the actual distilling surface may be depleted of the light constituents while the major portion of the distilland is still comparatively rich in the constituent desired. The pot type stills also require many hours of vacuum treatment to degas the material completely.

To avoid these disadvantages the falling-film still (Figure 5) came into being. This type still makes it possible to store the bulk of the material to be distilled at a low temperature and only exposes a small amount to heat. In

addition to decreasing the amount of decomposition obtained, this type still offers some assurance that all portions of the substance have an equally favorable opportunity for degassing and distillation.[7]

The falling-film still is one of the commercially-used continuous molecular stills where the material may be transferred over a succession of evaporators with each being at a higher temperature than the preceding one. In the form shown (Figure 5) the material is held in a container, A, and allowed

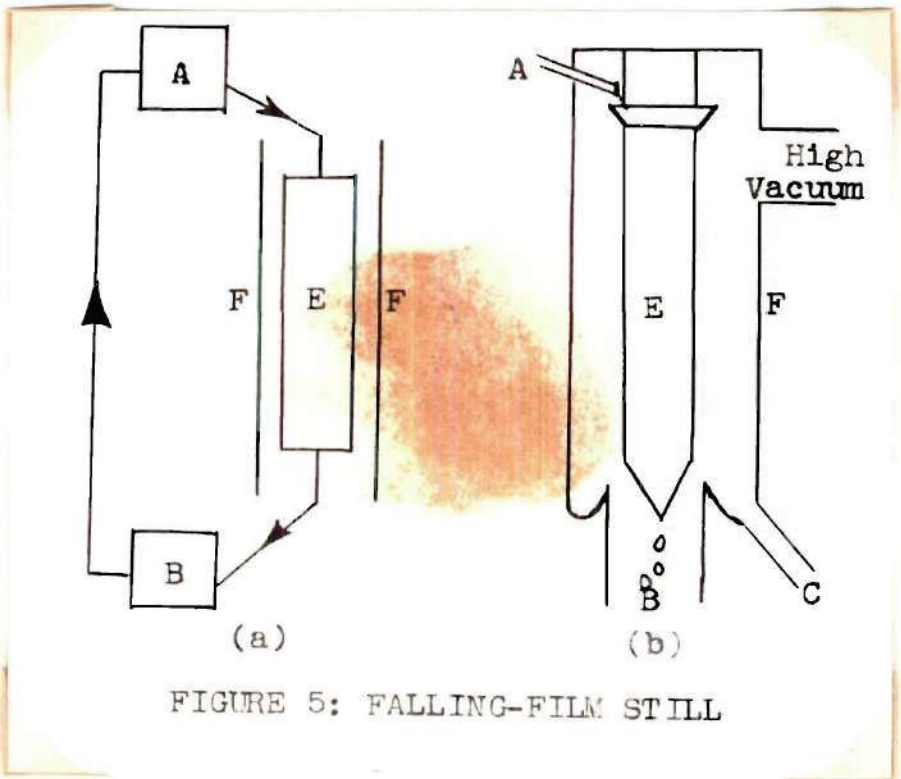


FIGURE 5: FALLING-FILM STILL

to flow down a heated column, E, into another container, B. The residue caught in B is recycled as shown in Figure 5 (a).

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[7]Hickman, K.C.D.; Industrial and Engineering Chemistry, 29, 968 (1937)

As the material passes over the heated column some diffusion takes place and the condensate from F is drawn off as product into a receiver at C.

The most critical part of the assembly is the distilling column which permits the distilland to flow in a thin, even stream over the heated surface. If the liquid forms channels or ripples while flowing, the thinner portions will be heated to a higher temperature and will be favored in the distillation. Thus, the thicker portions are neglected and the apparatus does not perform its proper function.

The search for a still which would have a large capacity with a low decomposition hazard led from the falling-film still to the centrifugal type still. This still consists basicly of a slightly conical rotor mounted on a shaft so that the cone can be rotated at 3000 to 5000 revolutions per minute. Oil is fed through a glass tube to the center of the rotating cone. The oil is then moved across the surface by centrifugal force and is collected in a rough of gutter located at the edge of the rotor.[8] The oil introduced on the centrifuge is slightly preheated and undergoes further heating on the conical surface. As oil flows over the surface diffusion takes places and a nearby condenser receives the distillate. Under these conditions the time of thermal exposure has been reduced from the few hours of the pot still to a small fraction of a second.

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[8]Hickman, K.C.D.; Science in Progress, 4th Series, Edited by G.A. Baitsell, Yale University Press, New Haven, Conn. (1945)

TABLE I [8]

TRANSITION FROM POT STILL TO CENTRIFUGAL STILL

Approximate Date	Type Still	Approx.* Distilland Thickness	Approx.** Molecular Thickness	Approx. Time of Exposure
1922	Laboratory Pot Still	1-5 cm	5×10^7	1-5 hr.
1935	Industrial Falling-Film Still	1-3 mm	5×10^5	2-10 min.
1930	Laboratory Falling-Film Still	0.1-0.3 mm	5×10^4	10-50 sec.
1940	Industrial Centrifugal Still	0.03-0.06 mm	10000	0.1-1 sec.
1936	Laboratory Centrifugal Still	0.01-0.02 mm	3000	0.04 - 0.08 sec.
1942	High-Speed Centrifugal Still	0.001-0.005 mm	400	0.001 - 0.005 sec.

* Assuming similar throughput for same unit area of all stills.

**Number of molecules, assuming molecular diameter of 15 Angstroms.

TABLE II [8]

RELATIVE THERMAL EXPOSURES

Pressure (mm Hg)	Temp. (°C)	Time (Sec)	Relative Decomposition	Type of Still
760	360	3600	1.5×10^{12}	Simple Flask
10	270	3600	3×10^9	Claissen Flask
1	220	3600	9.2×10^7	Wipe-Neck Pot Still
1	220	60	1.5×10^6	Petroleum Flask Still
.001	130	3600	1.8×10^5	Molecular Pot Still
.001	130	60	3000	Molecular Falling-Film Still
.001	130	1	50	Molecular Centrifugal Still
.001	130	.02	1	Molecular Centrifugal Still

As a matter of interest a comparison of exposure times and film thicknesses are shown in Table I and a comparison of probable thermal exposures in Table II.

From these tables it is clear that the centrifugal still offers a freedom from decomposition which may be more than actually needed. Since the time of exposure is so low the material may be heated above the decomposition temperature. This increase in temperature would in turn increase the rate of distillation and considering the amount of material passing over the surface a large capacity can be obtained with esentially no decomposition.

Figure 6 shows one basic type of centrifugal still.[9] A is the rotating evaporator, a cone with a 15° slope which thus embraces an angle of 150°. The distilland is fed to the center of the cone, A, by the tube, B, and it passes over the revolving cone to the stationery collection gutter at C from which it flows out into a reservoir at D. The revolving cone is covered by a glass bell jar, G, and the high vacuum is maintained by a vacuum line, L, partly hidden by the rotor, A. The bell jar can serve as a condenser or a separate condenser can be placed near the plate. In any event, the distillate collecting is removed at K. Auxiliary equipment for recycling and pumping is not shown since it is not an integral part of the still.

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[9] _____, "Information on High Vacuum Distillation", Distillation Products, Inc., Rochester, N.Y., (April 1947)

The stills discussed are all in use today with the centrifugal still being the most desirable from a general purpose view point. The others naturally have their own specific applications.

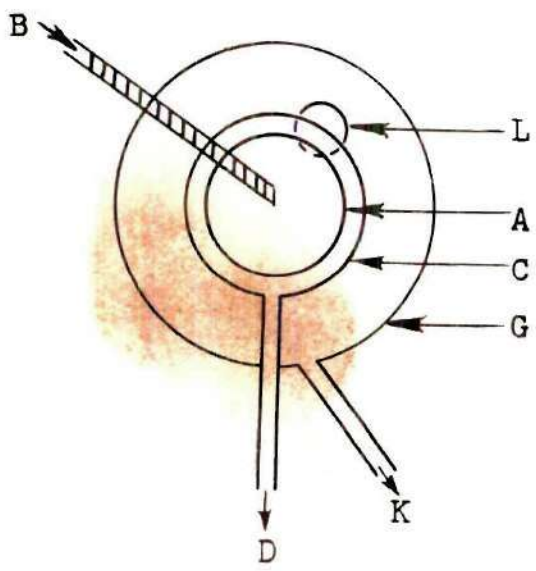

FIGURE 6: CENTRIFUGAL STILL

Further modifications of molecular stills can be found by referring to the bibliographies on molecular distillation compiled by Detwiler and Markley [10] and Detwiler,[11] which include some two hundred references. A complete review of

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[10] Detwiler, S.B. and Markley, K.S.; Oil and Soap Journal, 16, 2 (1939)

[11] Detwiler, S.B.; Oil and Soap Journal, 17, 241 (1939)

the history of molecular distillation and its scientific accomplishments may be found in an article entitled "High-Vacuum Short-path Distillation - A Review" by Hickman.[12]

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[12] Hickman, K.C.D.; Chemical Reviews, 34, 51 (1944)

THEORETICAL DISCUSSION

Elementary Theory

The rate of evaporation from a surface under high vacuum has been determined by Langmuir[13] from theoretical considerations based on the kinetic theory of gases.

$$m = p\sqrt{\frac{M}{2\pi RT}} \qquad (1)$$

m = rate (gms/sec/cm^2)
p = vapor pressure (dynes/cm^2)
M = molecular weight
T = temperature ($^\circ$Kelvin)
R = gas constant (83.2 x 10^6 ergs/$^\circ$K)

It should be pointed out that this equation applies only when the pressure in the system is reduced so low that the mean free path of the distilling molecules is of the same order of magnitude as the distance from the evaporator to the condenser. This should be approximately 0.5 to 10 cm. (In the still used in this problem it is 30 cm.)

The calculations of the mean free path should then be considered as an integral part of the preliminary work on any molecular distillation problem. Maxwell showed that, considering the distribution of velocities among molecules, an average value for the mean free path is found by Equation (2).

$$L = \frac{1}{\sqrt{2}N d^2 \pi} \qquad (2)$$

L = mean free path (cm)
N = molecules/cm^3
d = molecular diameter (cm)

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[13] Langmuir, I.; Physical Review, 2, 329 (1913)

Yarwood[14] has estimated the values of the mean free path of air molecules as shown in Table III. The molecular diameters of materials used in molecular distillation are greater than air molecules; and consequently the mean free path will be less. However, the table shown still gives an indication of the required vacuum.

TABLE III

MEAN FREE PATH OF AIR

Pressure (mm Hg)	Mean Free Path (cm)
760	10^{-5}
10^{-2}	1
10^{-3}	7
10^{-4}	70
10^{-5}	7000

Once the required high vacuum is obtained, evaporation takes place according to Langmuir's equation (1). The evaporation rate then becomes dependent upon the temperature and the molecular weights of the materials in the still. In passing, it should be mentioned that the actual distillation rate will be less than the evaporation rate because of various factors such as the shape of the apparatus and the distance

[14] Yarwood, J.; High Vacuum Technique, Chapman & Hall, Ltd., London (1948)

from the evaporator to the condenser. It may be noted at this point that there is no vapor-liquid equilibrium or anything corresponding to this in molecular distillation.

In a continuous still of the falling-film or centrifugal type the thickness of the film of liquid on the evaporator is also an influencing factor. The thickness in the falling-film still is dependent on the rate of flow and temperature of the liquid flowing over the evaporator surface. In the centrifugal still the thickness is dependent on these factors and also on the speed of rotation. Since the rate of evaporation is usually greater than the diffusion of the light molecules through the liquid in pot type stills, there is a tendency for the surface layer to become depleted of the volatile constituents. The falling-film and centrifugal stills are designed so that this condition is minimized.

The temperature difference of the condenser and evaporator plays an important role in molecular distillation (Similar to the surface temperature in radiant heat transfer). If the condenser temperature is too high evaporation will offset some of the condensation. The selection of the cooling medium then becomes an important factor. Washburn recognized this fact and presented the following equation which may be used in the selection of a cooling agent. It gives the minimum desired temperature difference between the condenser and the

evaporator for satisfactory molecular distillation.

$$\Delta t = \frac{T \log X}{\log X + \phi (T_B/T)} \qquad (3)$$

$$X = \frac{p_e}{p_c}$$

T = temperature (^{o}Kelvin)
T_B = normal boiling point
t = temperature (^{o}Centigrade)
p_c = vapor pressure at temperature of condensing surface
p_e = vapor pressure at temperature of evaporating surface
ϕ = constant from International Critical Tables, Vol. 3, pg. 246 (1910). (This constant is dependent on molecular weights, viscosities, and vapor pressures.)

In passing, it may be pointed out that azeotropic mixtures, which cannot be separated in ordinary types of stills, may be separated by molecular distillation; since from Langmuir's equation (1) it is evident that the separation will be determined not only by the partial pressures of the materials but also by their molecular weight. There is one point in molecular distillation which could be termed "an azeotropic condition". That is when the molecular ratio (n_1/n_2) is equal to the ratio $(p_1/p_2)\ (M_2/M_1)^{\frac{1}{2}}$. It is highly improbable that a mixture would be azeotropic under both ordinary distillation and molecular distillation conditions. Hence, practically every mixture should be subject to separation by one method or the other, or a combination of both.

Theory of Elimination

In addition to the basic theory outlined above, it is desirable to have an understanding of the elimination theory. The theory rests on simple postulates. Assuming that the substance to be distilled has no affinity for its solvent, the quantity evaporated is proportional to its mole concentration in the distilland. It can easily be seen that if a volatile substance were to be distilled from a non-volatile substance that the rate of elimination or distillation of the volatile constituent would constantly decrease and would finally approach zero (assuming the temperature remains constant). To increase the speed of this elimination the temperature must be increased. This increase would cause the elimination rate to become greater, and if the temperature were increased at intervals the elimination rate would reach a peak and then fall toward zero in spite of the increasing temperature. This is due to progressive removal of the volatile materials from the distilland.

Hickman[15] presented the following problem to illustrate the elimination theory. "Let the distillability be doubled every third step (the distilland being exposed for equal time intervals at each temperature and the temperature raised so

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[15]Hickman, K.C.D.; Industrial and Engineering Chemistry, 29, 971 (1937)

that the distillability is increased by an equal amount at each step. Let 1 per cent of the material be eliminated during the first interval." Simple arithmetic gives the trend of the elimination shown in Table IV. Figure 7 shows the plotted values.

TABLE IV

ELIMINATION DATA

Time Interval	Distillability	% Eliminated from Distilland	Rate of Elimination
1	1.26	2.25	1.25
2	1.59	3.80	1.55
3	2.0	5.72	1.92
4	2.52	8.09	2.36
5	3.18	11.00	2.88
6	4.00	14.56	3.51
7	5.23	18.87	4.23
8	6.36	24.04	5.04
9	8.00	30.11	5.91
10	10.06	37.15	6.80
11	12.67	46.10	7.65
12	16.00	54.73	8.36
13	20.15	63.86	8.80
14	25.04	73.03	8.83
15	32.00	81.67	8.36
16	40.70	89.16	7.35
17	50.4	95.62	5.88
18	64.0	98.42	4.18
19	80.7	---	2.57
20	101.61	---	1.30

By standardization of distillation methods it is possible to obtain a single elimination curve for the distillation

of any volatile substance from a non-volatile solvent. By use of these elimination curves it is possible to identify substances in much the same manner as chemical compounds are identified by such properties as their boiling points. The

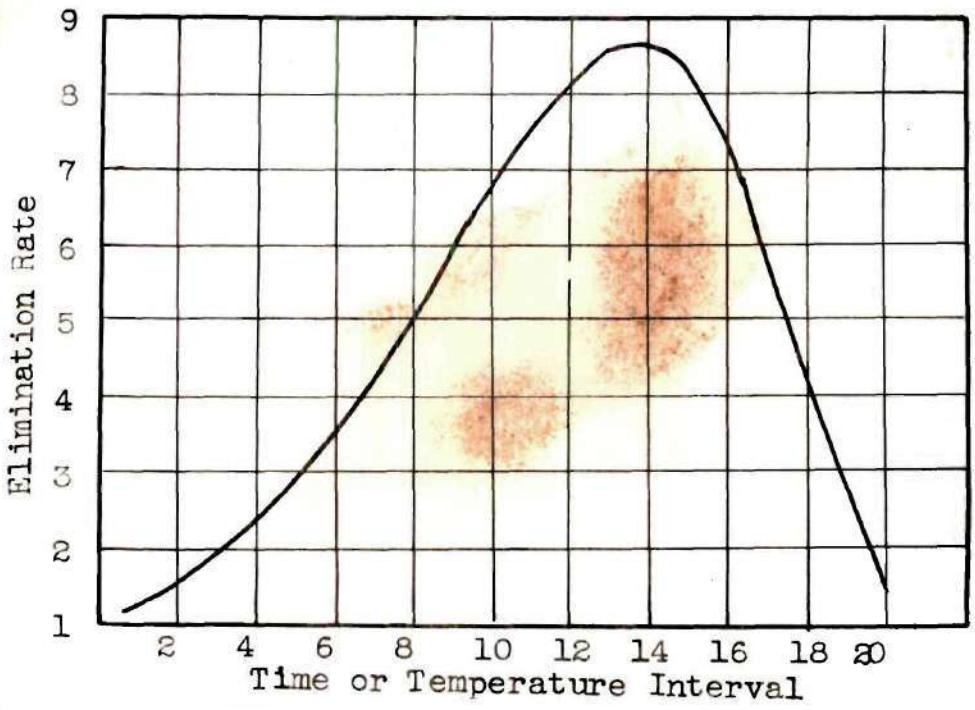

FIGURE 7; ELIMINATION CURVE

elimination curve is then a property of each individual material. Also from the shape and slope of the elimination curves approximate vapor pressure diagrams can be constructed to further aid in the study of the materials.

Embree[16] has obtained elimination curves by mathematical methods using certain physico-chemical principles. Namely:

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[16]Embree, N.D.; Industrial and Engineering Chemistry, 29, 975 (1937)

$$V = kND \qquad (4)$$

$$p = NP \qquad (5)$$

$$n = NpA\sqrt{\frac{1}{2\pi MRT}} \qquad (6)$$

V = rate of distillation
k = proportionality constant
N = concentration as mole fraction
D = distillability (ratio of number of molecules leaving a surface to the number remaining on the surface)
p = partial pressure
P = total pressure
n = Langmuir's rate
A = area
M = molecular weight
T = absolute temperature
R = gas constant

Using Equations (4), (5), and (6) Embree proved that the amount eliminated at any temperature during a specific time interval can be determined by Equation (7).

$$Q_{i+1} + Q_i = \int_0^S \frac{V_i}{100}\,ds \qquad (7)$$

$$\Delta Q = (1 - Q_i)\left(1 - e^{-D_i S/100}\right)$$

Q = fraction of original distilled
S = time
i = interval number

The fraction of the total amount distilled at each temperature may be calculated by Equation (7) and the results plotted versus temperature will give a curve similar to that obtained by Hickman (see Figure 7).

The elimination theory coupled with the basic concepts of the mean free path and Langmuir's equation give a complete theorectical working pattern of molecular distillation.

EQUIPMENT AND ACCESSORIES

The equipment used in this work consisted of a pot type still and column patterned after one suggested by Hickman and Weyerts,[17] a dual receiver system so designed as to permit continuous operation, a McLeod gauge attached to the top of the column, an oil diffusion pump, two mechanical pumps, a thermocouple-potentiometer arrangement for measurement of temperatures at desired points, two "Variacs" for heater control, and a magnetic stirrer for agitation of the distilland.

The still-pot was a 500 cc Eclenmeyer flask with a 50/30 socket joint at the neck. This flask was insulated with asbestos and two heating coils wound on the flask. The first heater, at the base of the flask, was the primary heater for the still; the second, on the sides of the flask, was to prevent heat losses. The column (Figure 8) consisted of a 30 mm diameter glass tube approximately 18 inches long with a 50/30 ball joint at the bottom, a 35/20 ball joint at the exhaust to the high-vacuum, and a 29/42 tapered joint at the top, onto which was connected the McLeod gauge and through which the internal thermocouple leads were brought. A side arm with a three-way stopcock near the base of the column served as the passage to the distillation receivers. The column was enlarged at the base to form an expansion bulb which

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[17] Hickman, K.C.D. and Weyerts, W.; Journal of American Chemical Society, 52, 4714 (1930)

was located below the condenser. The condenser was cooled by a jet of compressed air. (See Figures 9, 18 and 19).

Figure 8 shows the location of the heaters and thermocouples. Thermocouple #1 is located inside the still just above the evaporation surface; #2 is located inside the expansion bulb; #3 and #4 are in the insulation of the still and were used to determine whether the system was adiabatic or not. The thermocouple calibration curve is shown in Figure 16. The location of the heaters is self-explanatory from the diagram.

The receiver system was designed so that either of the two receivers could be evacuated without being joined into the distillation system and in this way one receiver could be in use while the other was being emptied. The arrangement used to do this may be seen in Figure 9. The actual receivers were 100 ml flasks having a 35/18 socket-joint neck to facilitate changing.

The remainder of the equipment was more or less standard. A McLeod gauge with a readable range down to less than 0.01 micron was used for determining pressure. The gauge used mercury as a fluid and was controlled by use of atmospheric pressure and vacuum pump. Figure 10 shows the calibration curve obtained.

One of the two mechanical pumps employed was a large

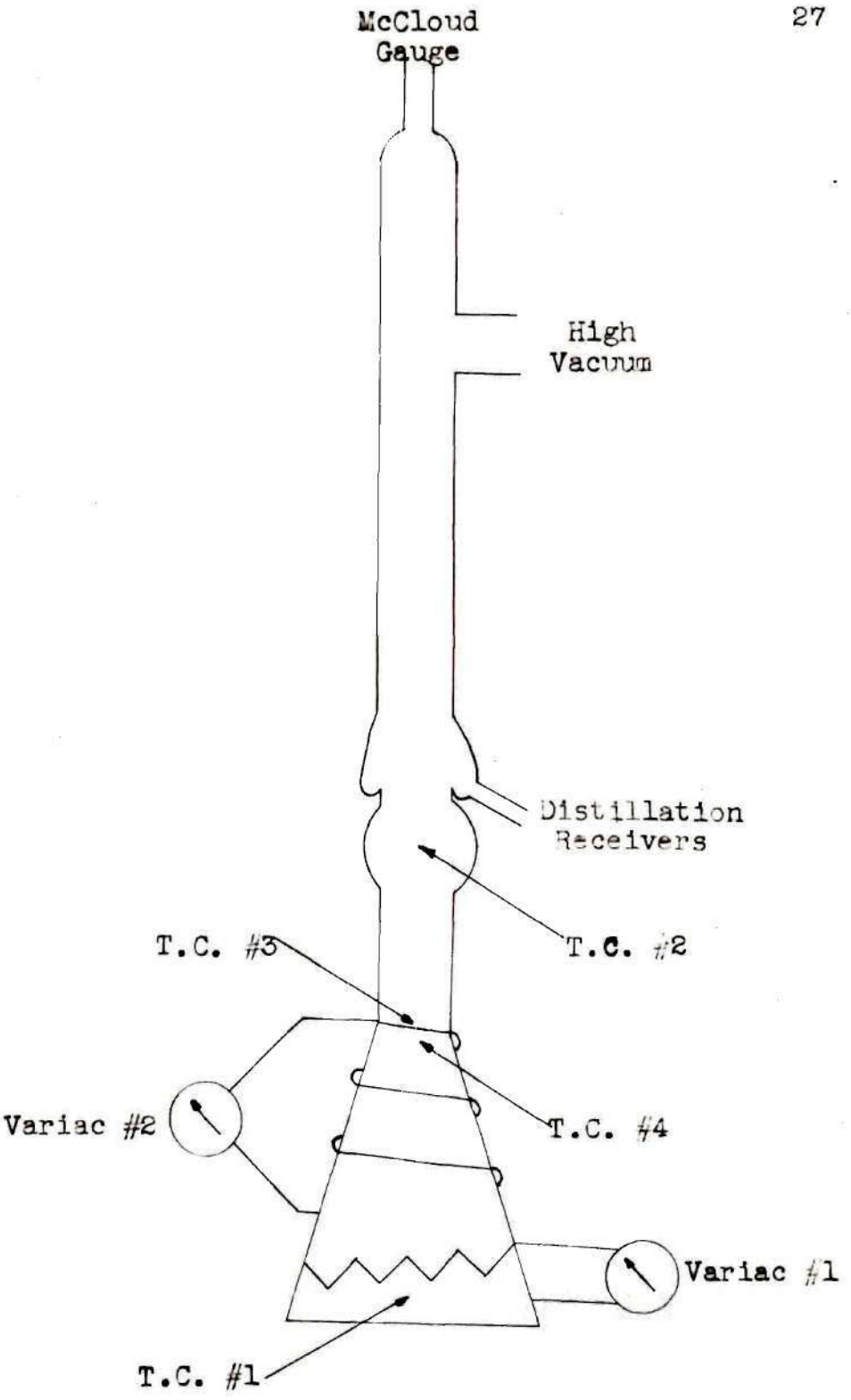

FIGURE 8: DISTILLATION SYSTEM
(Showing Thermocouple Locations)

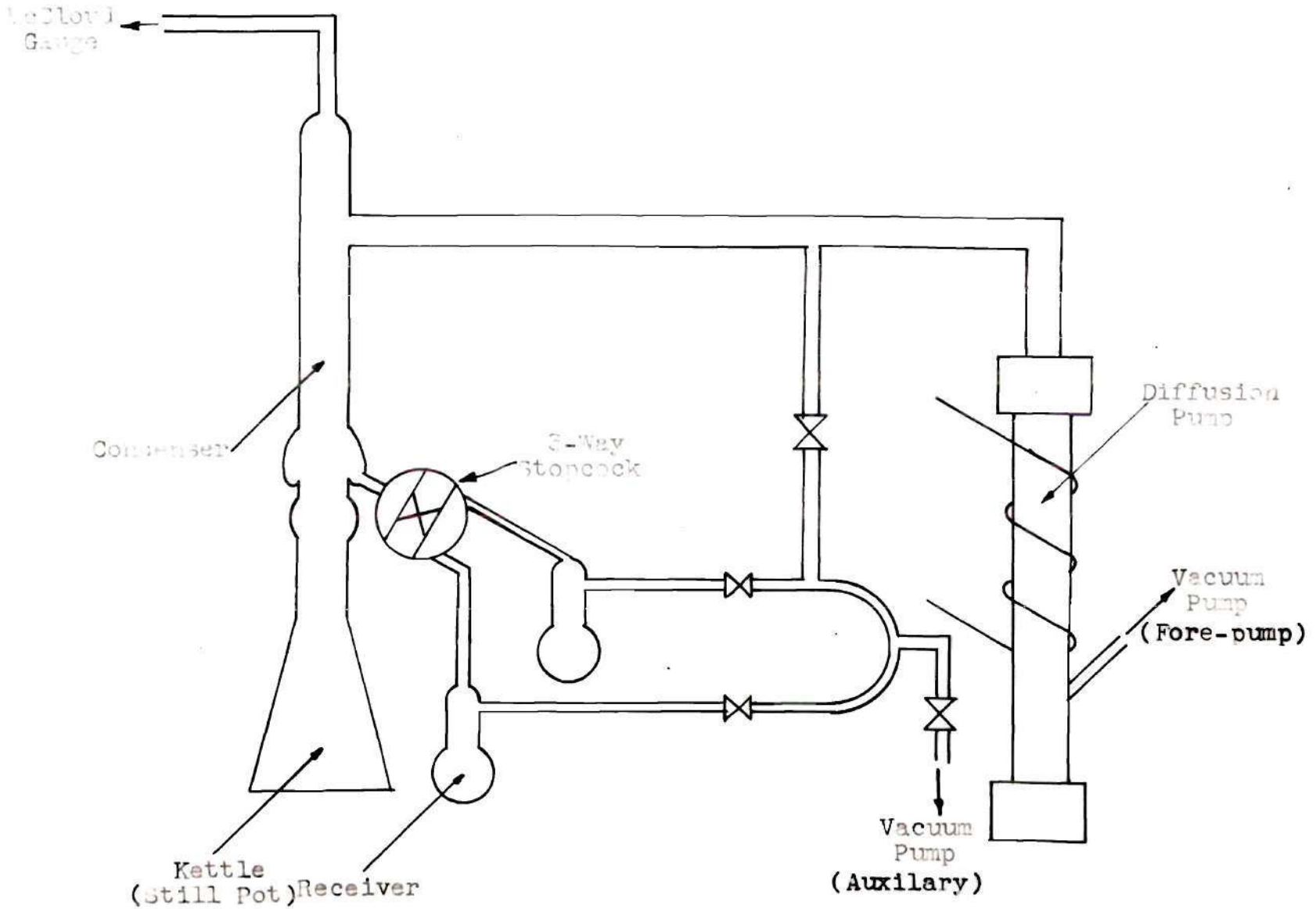

FIGURE9 : DIAGRAM OF EQUIPMENT

(1/2 H.P.) Cenco "Megavac" which was used as the forepump, and the other was a small (1/4 H.P.) Welch "Duo-Seal" which served as the auxiliary pump. The auxiliary pump was used to evacuate the receiving system and lower the mercury in the McLeod gauge.

An oil diffusion pump produced the high vacuum. This pump was a triple-jet diffusion pump with a normal operating pressure of 0.002 microns and a pumping speed of 50 liters per second. Details of operation and maintenance are given in Form 113 of the National Research Corporation.[18]

The magnetic stirrer consisted of a magnetized stirring bar which was placed inside the pot, and an external magnet which could be rotated at various speeds; since theinside magnet followed the rotation of the outside magnet the stirring could be controlled.

In addition it is necessary to have good waxes, greases, and oils for use in high vacuum systems. The greases and waxes must have a sufficiently low vapor pressure to prevent their contaminating the system. The viscosities must be such that their use is feasible on ground-glass joints and stopcocks. The grease used in this work was Dow-Corning's High-Vacuum Stopcock Grease with a reported vapor pressure of 10^{-6} mm Hg. This grease was found to be unsuitable.

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[18]National Research Council, Vacuum Engineering Division, Form 113 (2/21/49)

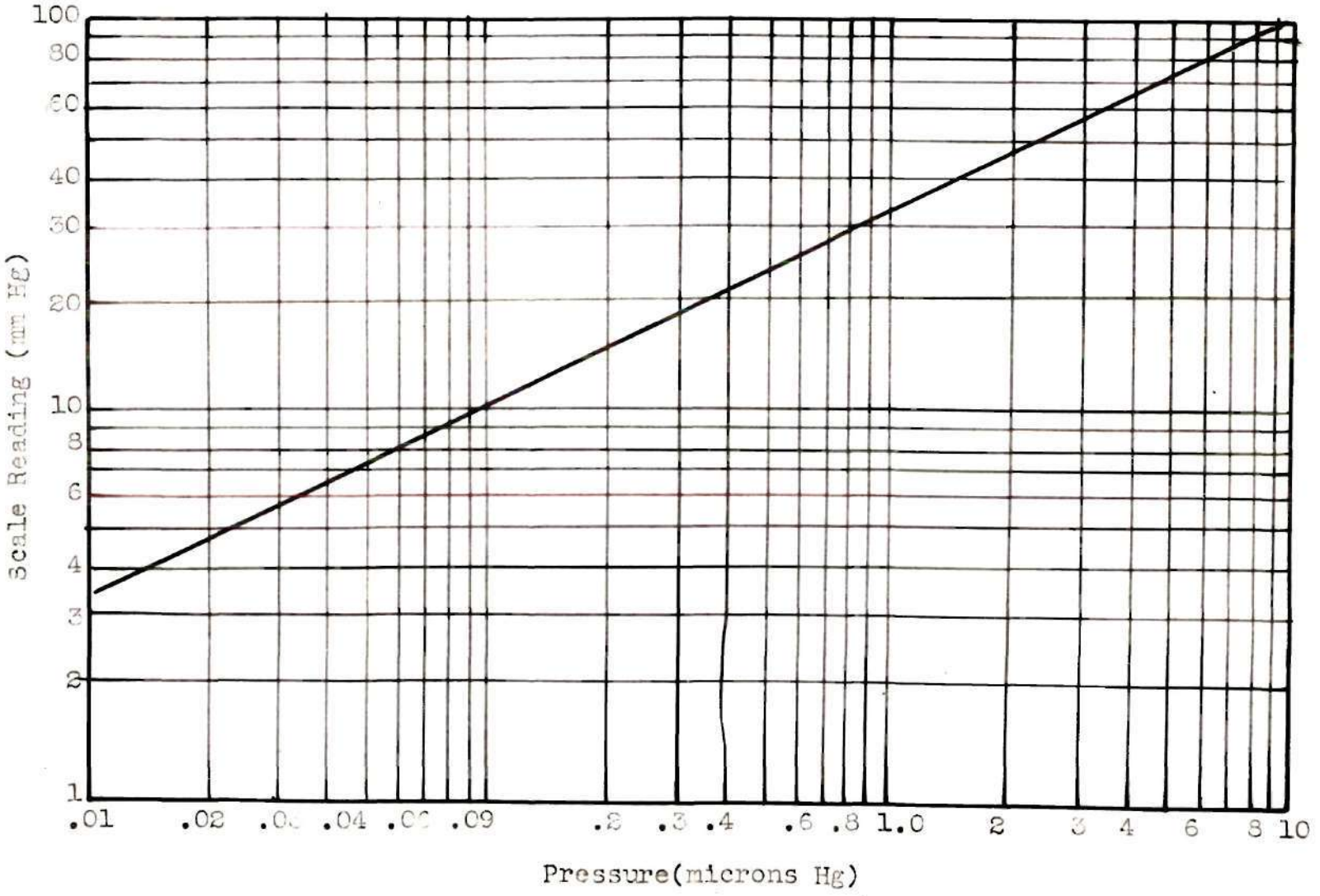

FIGURE 10: MC CLOUD GAUGE CALIBRATION

A comprehensive study concerning vacuum technique and equipment has been compiled by Hickman and published by the Franklin Institute.[19]

The McLeod gauge was calibrated by measuring the volume of the main bulb, which was done by filling the bulb with water and measuring the volume directly. This volume was found to be 298 cc. The capillary was then measured by filling a length with mercury. The capillary volume was found to be 2.6×10^{-4} cc/mm. Using these figures and the perfect gas law the curve shown in Figure 10 was obtained as follows:

$$P_1V_1 = P_2V_2 \qquad (8)$$

When the capillary length is 100 mm the compressed pressure may be assumed to be the same since the low system pressure may be neglected. Then P_1 100 mm, V_1 100 x 2.6 x 10^{-4} cc, and V_2 298 cc, and:

$$P_2 = \frac{100 \times 100 \times 2.6 \times 10^{-4}}{298}$$

$$P_2 = .0087 \text{ mm} = 9 \text{ microns}$$

By calculating several points and plotting on log-log paper, the calibration curve was obtained.

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[19]Hickman, K.C.D.; Journal of the Franklin Institute, 213, 119 (1932)

The thermocouples were calibrated by using ice at 0° C. and boiling water at 100° C. and plotting the potentiometer readings against temperature on a linear scale (Figure 16). All of the thermocouples were checked and one calibration curve held true for each.

EXPERIMENTAL PROCEDURE AND RESULTS

The procedure used was to charge the still with 200 cc of di-butyl pthalate and to evacuate the system to the lowest possible pressure before beginning a distillation run. The fore-pump was used to evacuate the system to a pressure of approximately 10 microns, at which point the diffusion pump was turned on and allowed to run until the pressure became constant at a minimum of 0.01 to 0.03 microns.

The distillation was then started by increasing the temperature of the pot. Pressure and temperatures readings were taken every fifteen minutes (recorded readings are at thirty minute intervals). The distillate was collected and the time of collection and the volume of each fraction noted.

After a number of fractions were collected at one temperature, the heat input was increased and additional fractions taken until the whole temperature range had been covered. The data presented do not have a larger spread because the still operation is extremely slow at temperatures below 120° C., and the sealing wax (DeKhotinsky cement) begins to melt at around 150° C causing leaks in the system and destroying the high vacuum.

Determinations of the effect of various degrees of stirring on the rate of distillation were made, and it was found that mechanical agitation had no noticeable effect on

the rate of distillation. However, mechanical agitation would tend to increase the degree of separation and the still efficiency if a multi-component system were being distilled. In any distillation the concentration of the more volatile constituents at the evaporating surface becomes less than in the main body of the liquid. Consequently, the rate of removal of the more volatile materials would be lower than if the surface layer had the same concentration as the main body. Mechanical agitation tends to renew the surface layer and keeps its concentration the same as the bulk of the liquid; thus increasing the degree of separation which would be obtained.

From the data taken and from Langmuir's equation (1) the actual rates of distillation and the theorectical rates were calculated, and from these items the efficiencies of the still were determined.

From Langmuir's equation it is possible to calculate the theoretical rate of evaporation (Based on a 64 cm^2 area) and this rate plotted against temperature (Figure 11) gives a basis for comparison of the experimental data. It should be pointed out that the 64 cm^2 area is the actual area of the evaporating surface. However, the cross-sectional area of the tube leading to the condenser is only 7 cm^2.

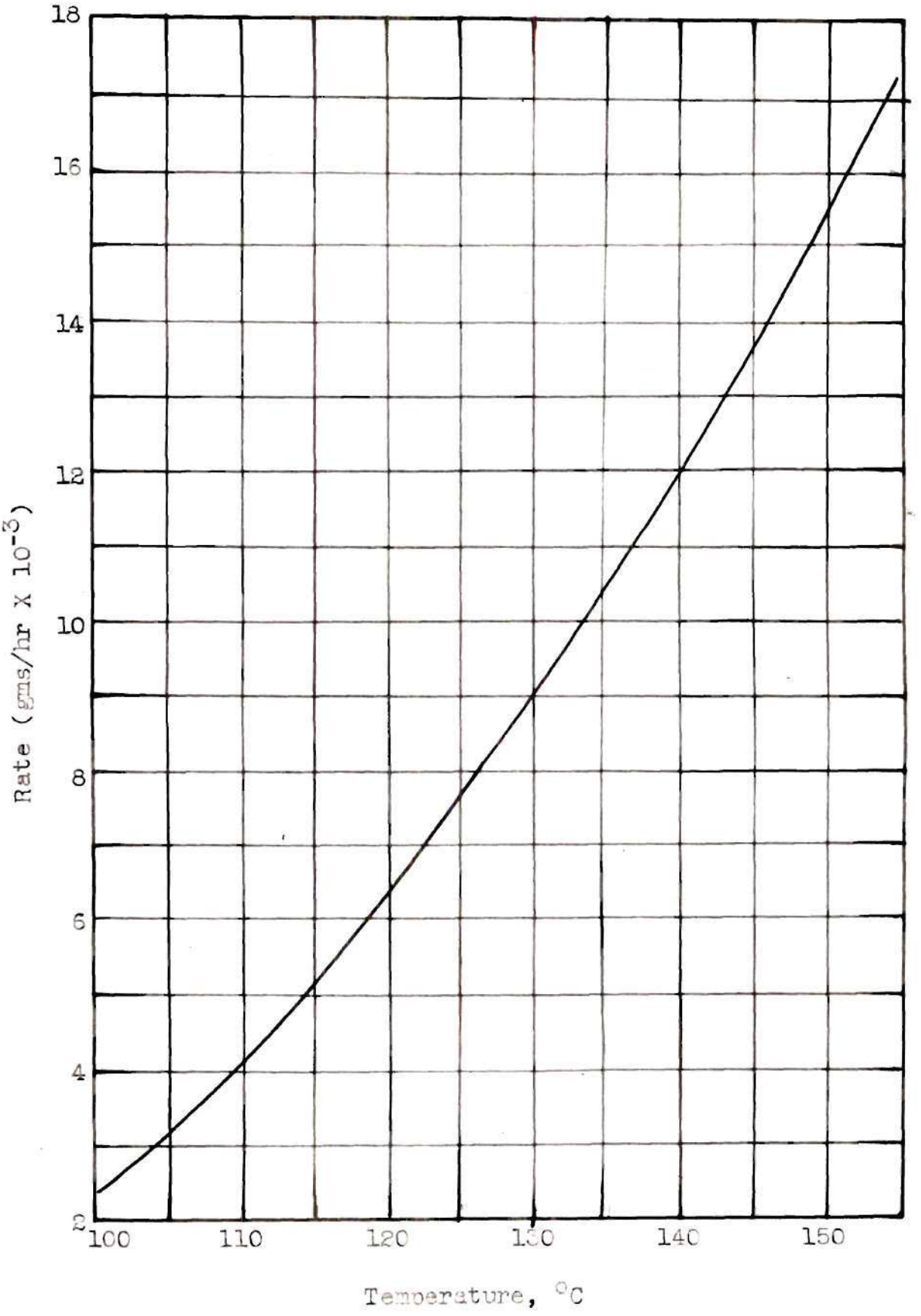

FIGURE 11: THEORETICAL RATE OF EVAPORATION

The following derivation was obtained from the kinetic theory of gases,[20] based on the evaporation from a surface of 1 cm^2 to another surface of 1 cm^2. (See Figure 12).

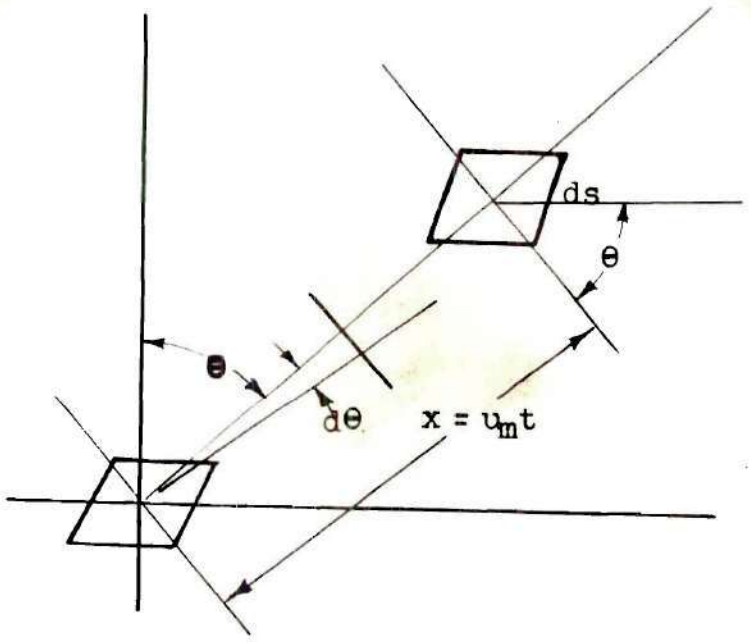

FIGURE12; MOLECULAR DEVIATIONS

The probability that a molecule leaving the surface at the origin will have a direction between the angles θ and θ + dθ is

$$\text{Probability} = \frac{\sin \theta d\theta}{2}$$

The number of molecules which will pass from one surface to the other and pass through a plane normal to a line connecting the surface centers is then

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[20] Jnanananda, S.; High Vacua, D. Van Nostrand Co., New York (1947)

$$dN = \frac{n \sin \theta d\theta}{2} - \cos\theta dS$$

This is true because the projection of the surface line dS, will be $\cos\theta dS$. The number passing through the plane and arriving at the receiving surface per unit area of surface is

$$\frac{dN}{ds} = \int dn_{\square} = n_{\square} = \frac{n}{2}\int \sin\theta\cos\theta d\theta u_m t$$

$$n_{\square} = 1/4\ nu_m t$$

Assuming unit time this becomes

$$n_{\square}^{1} = 1/4\ nu_m$$

$$n_{\square}^{1} = 1/4\ nL$$

θ = angle of molecular travel
S = receiving surface
N = total number of molecules
n = number molecules/cm^3
u_m = mean molecular velocity
L = mean free path

From this it can be seen that the amount of di-butyl pthalate passing into the tube should be 1/4 of the amount evaporating from the projection of the tube cross-sectional area on the evaporating surface, or (1/4)(7/64)(total amount evaporated), which is 2.73% of the total calculated evaporation by Langmuir's equation. This rests on the postulate that all molecules which leave the evaporation surface never return. Such, of course, is not the case; so the estimated

rate could be reduced another 50% to a maximum possible rate of 1.37% of the total theoretical evaporation. The side walls of the relatively long passage from the evaporator surface to the condenser surface would be expected to have a large effect due to reflection, deflection, and condensation. Actually the amount of material collected was found to be between 0.10 and 0.15% of the theoretical rate as calculated by Langmuir's equation. This is of the order of magnitude which was expected.

The actual distillation rate increases with temperature as shown by Figure 13 (Data from Table XII), but the efficiency of distillation decreases with increasing temperature as shown in Figure 14. The randomness of the points is probably due to errors in the measurement of the distillate volumes and the variation in the performance of the condenser. The condenser action is probably the major source of error since it was noted that the droplets of condensate often appeared on the sides of the condenser to varying heights. These droplets were large enough to affect the actual sample volume by as much as 1 cc. which could cause an error in the efficiencies of 10% of the actual calculated values. Since an air-cooled condenser was employed the variation of its performance was partly due to changes in the room temperature, or air circulation in the room, or both.

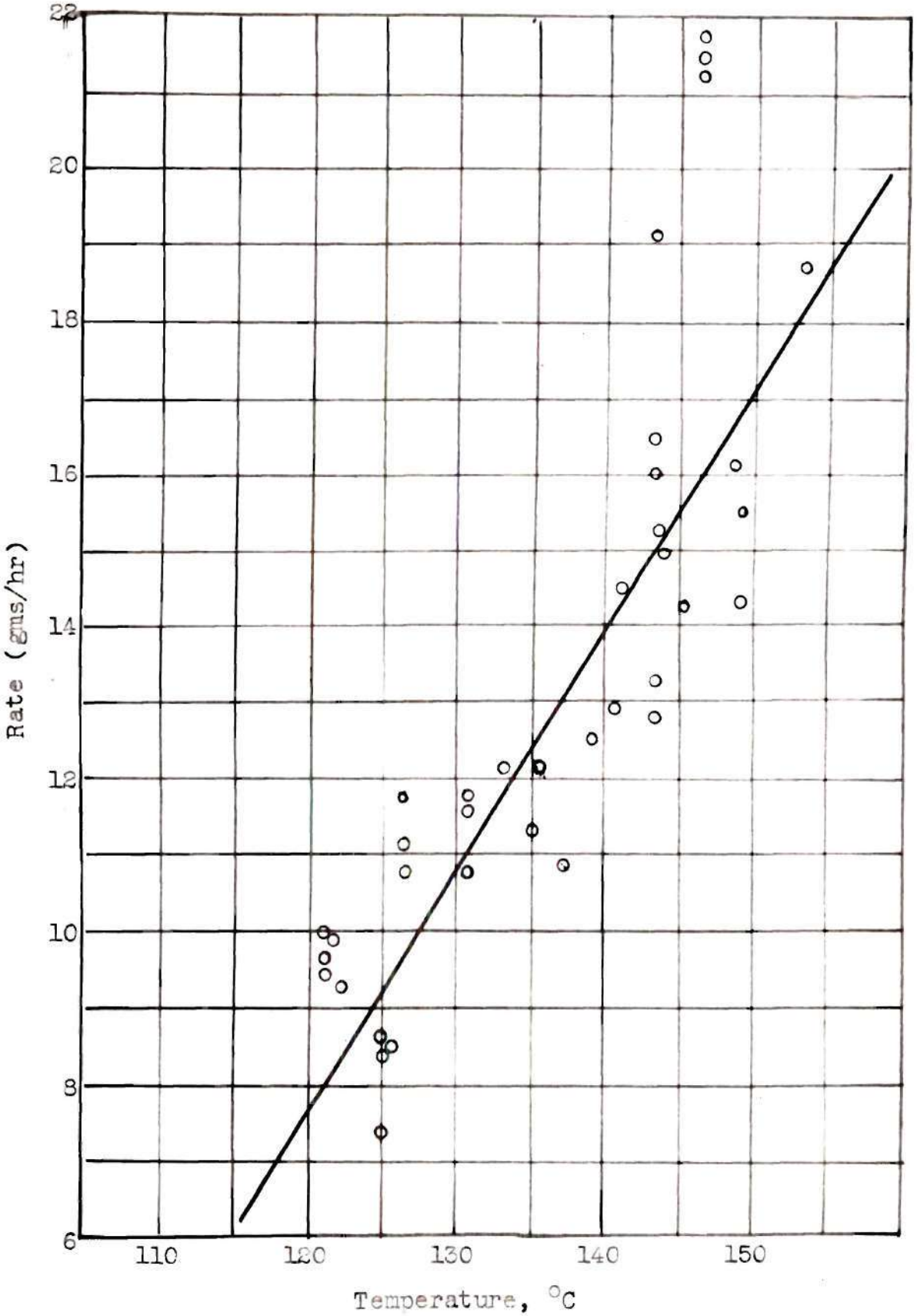

FIGURE 13: DISTILLATION RATE vs. TEMPERATURE

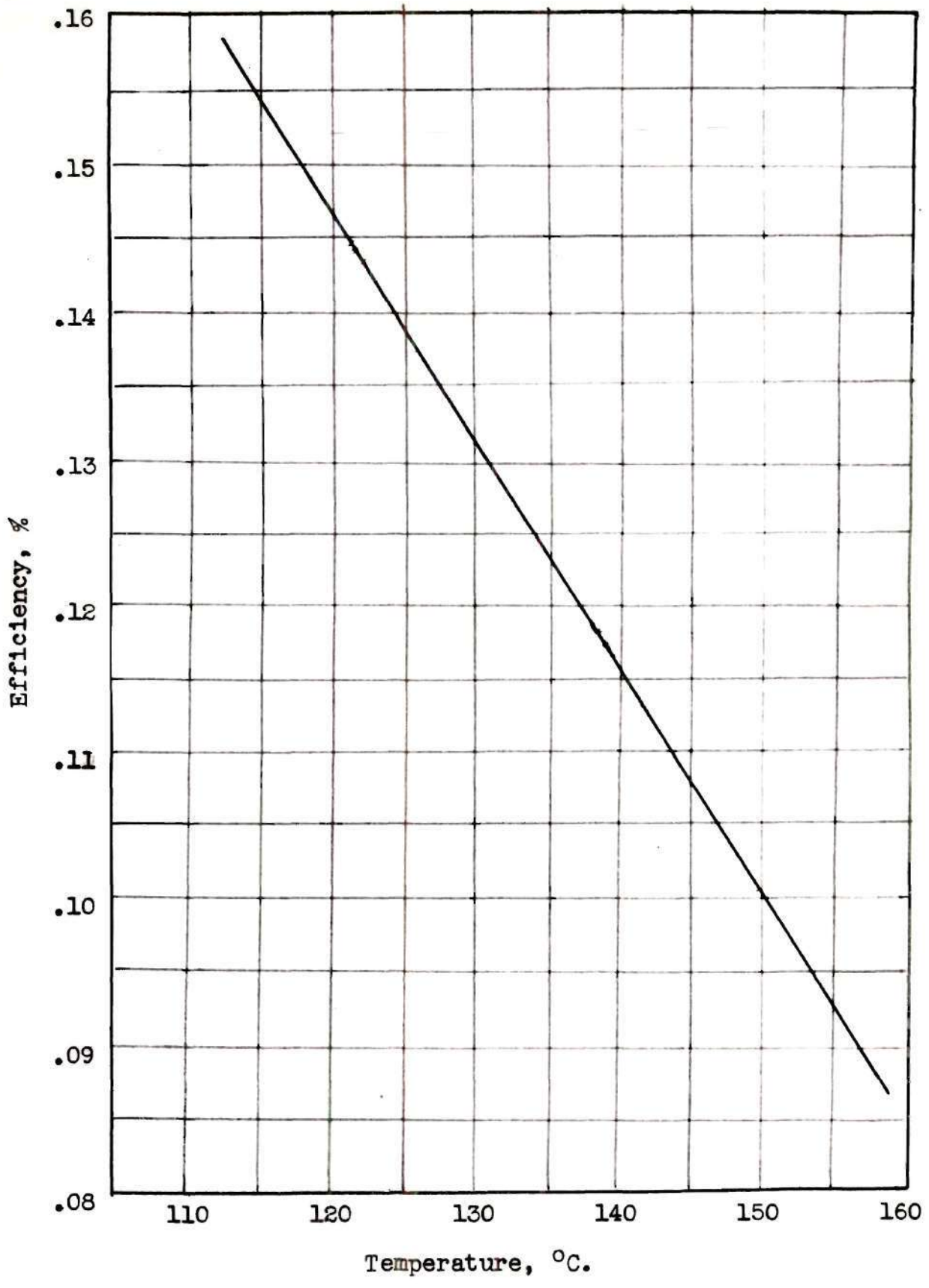

FIGURE 14: EFFICIENCY VS. TEMPERATURE

The efficiency plotted in Figure 14 as a function of temperature and in Figure 15 as a function of pressure. Data used in calculating rates and efficiency can be found in Appendix II.

From calculations based on theoretical concepts (see section on Methods of Calculation) it was found that the maximum allowable pressure ("critical pressure") for the molecular distillation of di-butyl pthalate is of the order of magnitude of 0.03 microns for the still used in this investigation. In Figure 15 it can be seen that the efficiency changes suddenly at this "critical pressure:. It is noted that at 0.03 microns the spread of points is greater than at any other pressure indicating the imminence of a discontinuity -- a shift in the conditions of operation. The fact that the rate of distillation changes by a factor of three at the calculated "critical pressure" (0.03 microns) shows that the nature of the distillation undergoes a definite change at this point.

In general the curves and data presented here indicate that the rate of distillation is only a small fraction of the theoretical rate obtained from Langmuir's equation. This discrepancy can be explained by several factors. As has been previously pointed out the distance from evaporator to condenser used by previous investigators was 0.5 to 10 cm; this

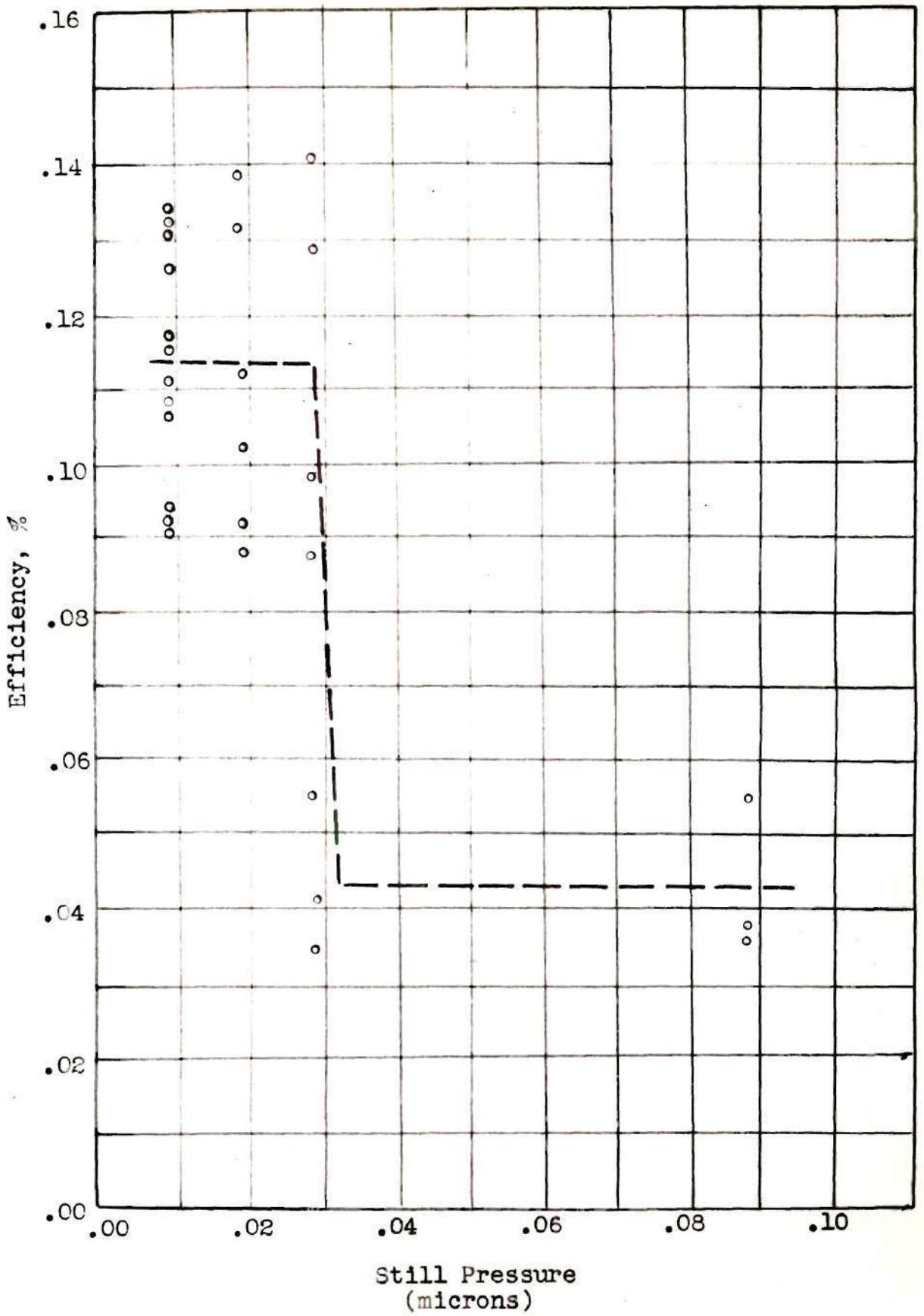

FIGURE 15: EFFICIENCY VS. PRESSURE

distance in the apparatus used in this investigation was around 30 cm. Even through the mean free path of the distilling molecules was great enough to reach the condenser, deflection and condensation by the tube leading to the condenser and the sides of the still pot prevented a truly efficient distillation from taking place. Then, too, as has already been pointed out, the "utilizable area" was much less than the "actual area" of the evaporating surface. The results are in accordance with the observed limitation of the molecular distillation technique discussed in previous paragraphs.

METHODS OF CALCULATION

Pauling[21] outlined a method for calculating the diameter of molecules using bond distances and atomic radii. Using this method and assuming the di-butyl pthalate molecule to be planar, the largest possible diameter is 12 Angstroms. Since this molecule is not planar and since the butyl side chains have an angle of about 110° between carbon atoms and these angles may be in any of an infinite number of directions the maximum diameter was taken to be 10 Angstroms and this figure used as the maximum molecular diameter of di-butyl pthalate molecules.

Using this diameter in an empirical equation (9) suggested by Dushman[22] the maximum pressure which could exist in the still was calculated.

$$L = \frac{2.331 \times 10^{-20} T}{P\, d^2} \quad \text{cm} \qquad (9)$$

$$P = \frac{2.331 \times 10^{-20} T}{L\, d^2}$$

P = pressure (mm Hg)
d = molecular diameter (cm)
L = mean free path (cm)

Since the distance from evaporator to condenser is about 30 cm the mean free path, L, must be at least 30 cm. Assuming

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[21] Pauling, L.C.; Nature of the Chemical Bond, Cornell University Press; Ithaca, New York (1940)

[22] Dushman, S.; Vacuum Technique, John Wiley & Sons, New York (1949)

a temperature of 127° C this gives

$$P = \frac{2.331 \times 10^{-20} \times 400}{30\ (10 \times 10^{-8})2}$$

$$P = .000031 \text{ mm or } .03 \text{ microns}$$

In calculating the theoretical rates of distillation, Langmuir's equation (1) was used in the following form:

$$W = 3510 p A \sqrt{\frac{1}{T}} \qquad (10)$$

A = evaporation area (cm^2)
W = rate (gms/hour)
p = vapor pressure (mm Hg)
T = temperature (°Kelvin)

This equation was used to calculate the rates. A sample calculation is presented using Reading Number B-12 from Table VII.

$$W = 3510\ (0.7)(64)\sqrt{\frac{1}{398}}$$

$$W = 7900 \text{ gms/hour}$$

The actual rates were calculated by

$$W = \frac{\text{Vol. of fraction (sp.gr.)}}{\text{(minutes)}} \times 60 \qquad (11)$$

$$W = \frac{(8.1)\ (1.048)}{(60)}\ (60) = 8.5 \text{ gms/hr.}$$

Using equations (10) and (11) the efficiency was obtained.

$$\text{Efficiency} = \frac{8.5}{7900} \times 100 = 0.108\%$$

All data used for the calculations from which the curves of Figures 13, 14, and 15 were drawn can be found in Tables VI, VII, VIII, IX, X, XI, and XII.

CONCLUSIONS

From the results obtained in this investigation the following conclusions may be drawn.

(1) The rate of distillation increases as the temperature increases. (Figure 13).

(2) The still efficiency decreases as the temperature increases. (Figure 14)

(3) The "critical pressure" calculated for the molecular distillation of di-butyl pthalate in the apparatus used in this investigation is 0.03 microns.

(4) The measured "critical pressure" (0.03 microns) substantiates the theoretically calculated pressure. (Figure 15)

(5) Mechanical agitation has no appreciable effect on the distillation rate of a one component system.

(6) The rates obtained in this investigation (0.054 mols/hr) are in substantial agreement with the rates obtained by Washburn[5] (0.04 mols/hr) and Hickman and Sanford[23] (0.053 mols/hr) even though the distance from the evaporator to the condenser used in this investigation (30 cm) was much greater than that used by previous investigators (0.5 to 10 cm). This fact refutes Washburn's statement concerning actual distillation rates (see Historical Development).

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[23]Hickman, K.C.D. and Sanford, C.R.; Journal of Physical Chemistry, 34, 637 (1930)

(7) The rates of distillation obtained in this work were between 0.10 and 0.15% of the theoretical rates as calculated by Langmuir's equation. This is of the order of magnitude which was expected.

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APPENDIX I

(CURVES)

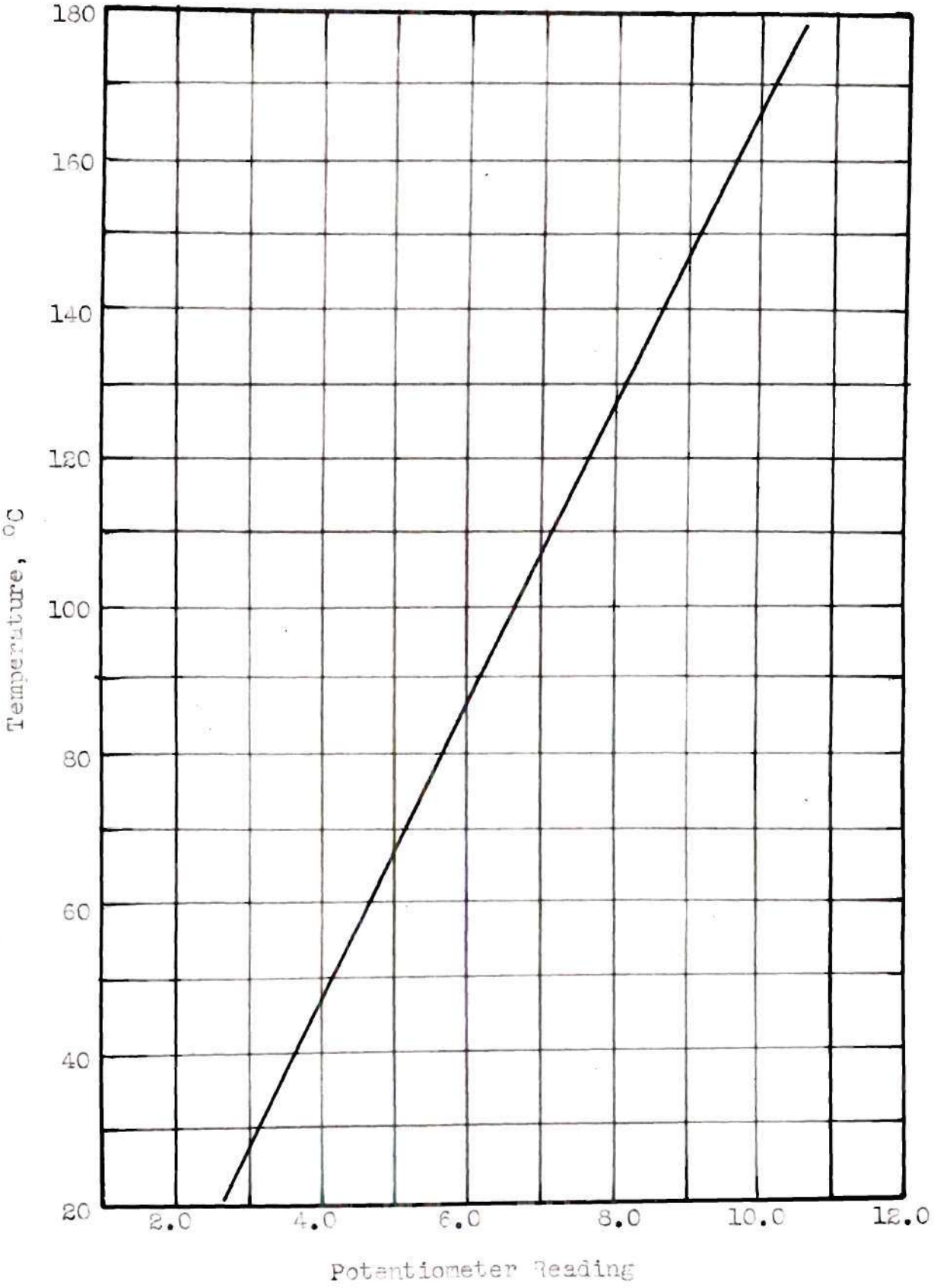

FIGURE 16: THERMOCOUPLE CALIBRATION

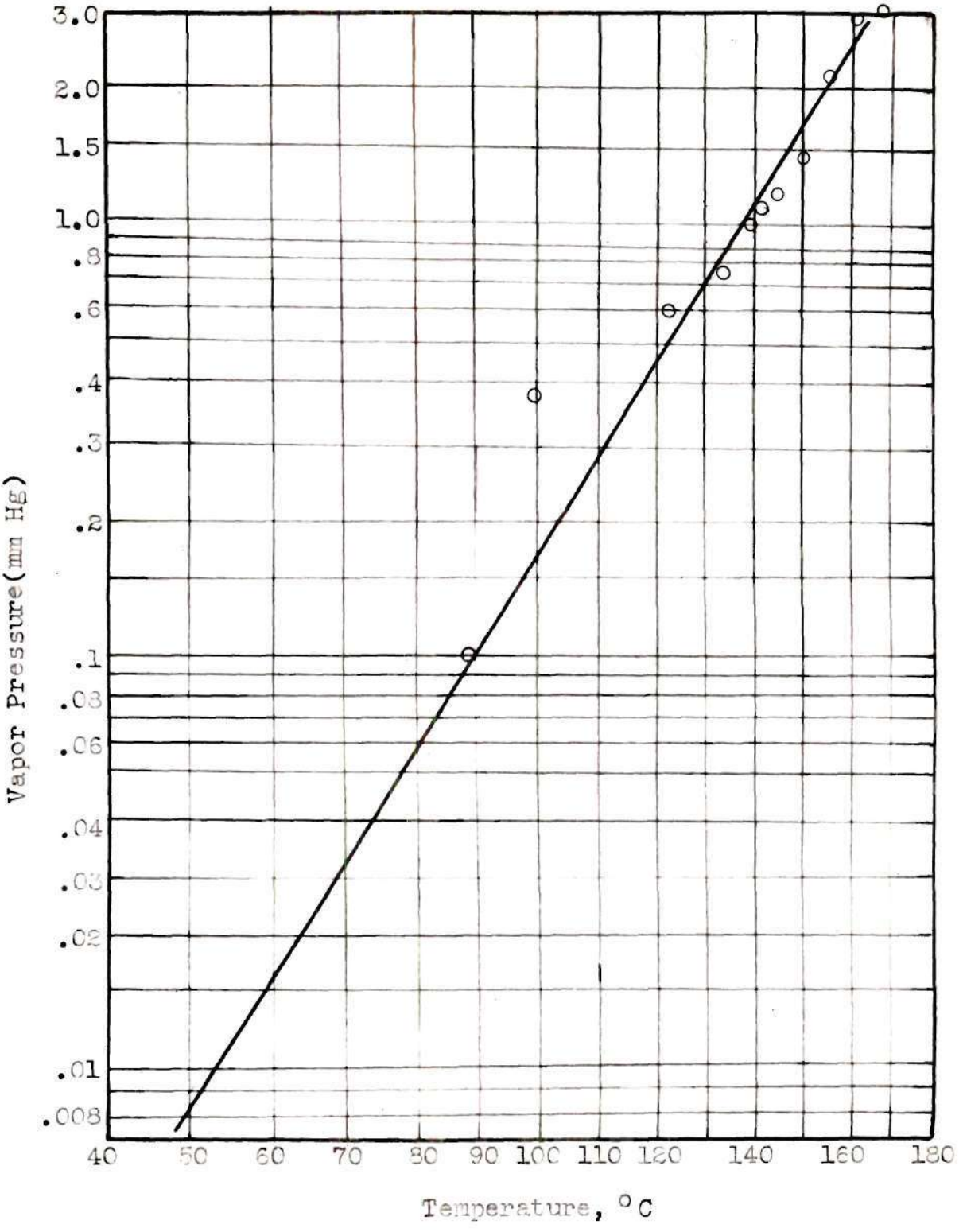

FIGURE 17: VAPOR PRESSURE OF BUTYL PTHLATE

APPENDIX II

(TABLES)

TABLE V

VAPOR PRESSURE OF DI-BUTYL PTHALATE[24]
(Used for plotting Figure 17)

Temperature (°C)	Vapor Pressure (mm Hg)
89.0	0.1
98.8	0.4
122.5	0.6
132.0	0.8
134.5	0.9
137.2	1.0
138.0	1.0
141.0	1.1
141.2	1.1
144.8	1.2
145.8	1.1
147.5	1.3
148.0	1.2
149.5	1.3
150.0	1.4
151.0	1.3
153.5	1.5
162.9	2.1
164.4	2.2
167.2	2.8
168.0	2.8
168.6	2.9
171.8	3.2
176.3	4.1

[24]Gardner, G.S. and Brewer, J.E.; Industrial and Engineering Chemistry, 29, 179 (1937)

TABLE VI: EXPERIMENTAL DATA (RUN A)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(c c)	Distillation Time (min)
A-1	.01	7.6	3.5	-	-	117	36	-	-	40	-		
A-2	.01	7.7	4.7	-	-	120	60	-	-	40	-		
A-3	.01	7.8	4.7	-	-	122	60	-	-	40	-		
A-4	.01	7.8	4.9	-	-	122	64	-	-	40	-		
A-5	.01	7.8	5.1	-	-	122	68	-	-	40	-		
A-6	.01	7.8	5.1	-	-	122	68	-	-	40	-		
A-7	.01	7.8	5.1	-	-	122	68	-	-	40	-		
A-8	.01	7.8	5.1	-	-	122	68	-	-	40	-	13.5	85
A-9	.01	7.8	5.1	-	-	122	68	-	-	40	-		
A-10	.01	7.8	5.1	-	-	122	68	-	-	40	-	8.7	55
A-11	.01	7.8	5.4	-	-	122	73	-	-	40	-		
A-12	.01	7.8	5.4	-	-	122	73	-	-	40	-	6.3	40
A-13	.01	7.8	5.4	-	-	122	73	-	-	40	-	6.2	40
A-14	.01	7.8	5.4	-	-	122	73	-	-	40	-		
A-15	.01	7.8	5.4	-	-	122	73	-	-	40	-	6.0	40
A-16	.01	7.8	5.4	-	-	122	73	-	-	40	-		

TABLE VII: EXPERIMENTAL DATA (RUN B)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
B-1	.02	7.0	6.4	6.4	6.4	105	93	93	93	30	10		
B-2	.02	8.2	7.3	7.3	7.2	129	111	111	110	30	12		
B-3	.01	8.2	7.6	7.6	7.4	129	118	118	113	30	12		
B-4	.01	8.1	7.6	7.5	7.4	127	118	116	113	30	12	15.7	60
B-5	.01	8.0	7.5	7.4	7.3	125	116	113	111	30	12		
B-6	.01	8.0	7.5	7.4	7.3	125	116	113	111	30	12	8.8	60
B-7	.01	8.0	7.4	7.3	7.2	125	113	111	110	30	12		
B-8	.01	8.0	7.4	7.3	7.2	125	113	111	110	30	12	8.3	60
B-9	.02	8.0	7.4	7.3	7.2	125	113	111	110	30	12		
B-10	.01	8.0	7.6	7.5	7.4	125	118	116	113	30	12	8.3	60
B-11	.02	8.0	7.5	7.4	7.3	125	116	113	111	30	12		
B-12	.01	8.0	7.5	7.4	7.3	125	116	113	111	30	12	8.1	60
B-13	.01	8.0	7.5	7/4	7.3	125	116	113	111	30	12		
B-14	.01	8.0	7.5	7.4	7.3	125	116	113	111	30	12	8.0	60
B-15	.02	8.0	7.4	7.3	7.3	125	113	111	111	30	12		
B-16	.01	8.0	7.4	7.3	7.3	125	113	111	111	30	12	8.1	60

TABLE VIII: EXPERIMENTAL DATA (RUN C)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
C-1	.02	8.0	7.9	7.9	7.8	125	123	123	122	30	12		
C-2	.02	7.9	7.9	7.8	7.7	123	123	122	120	30	12	4.4	60
C-3	.02	7.9	7.9	7.9	7.8	123	123	123	122	30	12		
C-4	.03	7.9	7.9	7.9	7.8	123	123	123	122	30	12	3.4	60
C-5	.03	7.9	7.9	7.9	7.8	123	123	123	122	30	12		
C-6	.03	7.9	7.9	7.9	7.8	123	123	123	122	30	12	3.9	60
C-7	.01	7.9	7.9	7.9	7.7	123	123	123	120	30	12		
C-8	.03	8.0	7.9	7.9	7.7	125	123	123	120	30	12	4.8	60
C-9	.02	8.0	7.9	7.9	7.8	125	123	123	122	30	12		
C-10	.02	8.0	7.9	7.9	7.8	125	123	123	122	30	12	7.0	60
C-11	.02	8.1	8.0	8.0	7.9	127	125	125	123	30	12		
C-12	.02	8.1	8.0	8.0	7.8	127	125	125	122	30	12	7.5	40
C-13	.02	8.1	8.0	8.0	7.9	127	125	125	123	30	12		
C-14	.02	8.1	8.0	8.0	7.9	127	125	125	123	30	12	12.4	70
C-15	.02	8.1	8.0	8.0	7.9	127	125	125	123	30	12		
C-16	.01	8.1	8.0	8.0	7.9	127	125	125	123	30	12	6.6	40

TABLE IX: EXPERIMENTAL DATA (RUN D)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
D-1	.01	7.9	7.8	7.8	7.7	123	122	122	120	35	12		
D-2	.02	8.1	8.1	8.0	7.8	127	127	125	122	35	12		
D-3	.02	8.2	8.2	8.2	8.1	129	129	129	127	35	12		
D-4	.01	8.3	8.3	8.3	8.2	131	131	131	129	35	12		
D-5	.01	8.3	8.3	8.3	8.2	131	131	131	129	35	12	7.7	45
D-6	.01	8.3	8.3	8.3	8.2	131	131	131	129	35	12		
D-7	.01	8.3	8.3	8.3	8.2	131	131	131	129	35	12	7.4	40
D-8	.01	8.3	8.3	8.3	8.2	131	131	131	129	35	12		
D-9	.01	8.3	8.3	8.3	8.3	131	131	131	131	35	12	7.5	40
D-10	.01	8.4	8.4	8.4	8.4	133	133	133	133	35	12		
D-11	.01	8.4	8.4	8.4	8.2	133	133	133	129	35	12	17.2	40
D-12	.01	8.4	8.4	8.4	8.3	133	133	133	131	35	12	11.5	30
D-13	.01	8.4	8.4	8.4	8.3	133	133	133	131	35	12	10.4	30
D-14	.01	8.4	8.4	8.4	8.4	133	133	133	133	35	12		
D-15	.01	8.4	8.4	8.4	8.4	133	133	133	133	35	12	28.4	80
D-16	.01	8.4	8.4	8.4	8.4	133	133	133	133	35	12		

TABLE X: EXPERIMENTAL DATA (RUN E)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
E-1	.09	8.9	8.5	8.5	7.6	143	136	136	118	32	12		
E-2	.09	8.9	8.5	8.5	7.6	143	136	136	118	32	12	7.5	70
E-3	.03	8.9	8.7	8.6	7.5	143	140	138	116	35	12		
E-4	.03	8.9	8.5	8.5	7.4	143	136	136	115	35	12	22.2	50
E-5	.03	9.1	8.7	8.6	7.5	147	140	138	116	35	12	10.1	30
E-6	.03	9.1	8.6	8.6	7.6	147	138	138	118	35	12	11.0	30
E-7	.03	9.1	8.6	8.6	7.6	147	138	138	118	35	12	9.0	25
E-8	.09	9.1	8.6	8.6	7.6	147	138	138	118	35	12		
E-9	.09	9.2	8.7	8.6	7.6	149	140	138	118	35	12	11.3	60
E-10	.03	9.2	8.6	8.6	7.6	149	138	138	118	35	12		
E-11	.03	9.2	8.6	8.6	7.6	149	138	138	118	35	12	15.3	60
E-12	.09	9.3	8.7	8.6	7.6	151	140	138	118	35	12		
E-13	.09	9.3	8.7	8.7	7.6	151	140	140	118	35	12	8.7	60
E-14	.03	9.0	8.5	8.5	8.4	145	135	135	113	0	0		
E-15	.01	9.0	8.5	8.5	7.4	145	135	135	113	32	12		
E-16	.01	9.2	8.5	8.5	7.4	149	135	135	113	32	12	11.0	45

TABLE X: EXPERIMENTAL DATA (RUN E), continued

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
E-17	.01	9.2	8.5	8.5	7.4	149	135	135	113	32	12		
E-18	.01	9.2	8.5	8.5	7.4	149	135	135	113	32	12	11.4	45
E-19	.01	9.0	8.5	8.5	7.4	145	135	135	113	32	12		
E-20	.02	8.8	8.5	8.3	7.4	141	135	131	113	32	12	9.3	45
E-21	.02	8.4	7.6	7.8	7.2	133	118	121	109	28	12		
E-22	.02	8.4	7.6	7.8	7.2	133	118	121	109	28	12	8.6	45
E-23	.02	8.5	7.8	7.9	7.2	135	121	123	109	28	12		
E-24	.01	8.6	7.9	8.2	7.2	137	123	129	109	28	12	7.7	45
E-25	.02	8.6	8.0	8.2	7.2	137	125	129	109	28	12		
E-26	.02	8.5	8.0	8.2	7.2	135	125	129	109	28	12	14.5	80
E-27	.01	9.4	8.7	9.0	8.1	153	140	145	123	40	12	9.0	30

TABLE XI: EXPERIMENTAL DATA (RUN F)

Reading Number	Pressure (microns)	Potentiometer Reading #1	#2	#3	#4	Temperature, °C #1	#2	#3	#4	Voltage #1	#2	Sample Vol.(cc)	Distillation Time (min)
F-1	.01	8.9	8.1	8.5	7.3	143	128	135	111	32	14	4.6	15
F-2	.01	8.9	8.3	8.6	7.5	143	131	138	115	32	14	7.9	30
F-3	.01	8.9	8.2	8.4	7.4	143	130	134	113	32	14	7.2	30
F-4	.01	8.9	8.3	8.6	7.5	143	131	138	115	32	14		
F-5	.02	8.9	8.4	8.6	7.5	143	133	138	115	32	14	7.2	30
F-6	.01	8.9	8.3	8.6	7.5	143	131	138	115	32	14		
F-7	.01	8.9	8.2	8.4	7.4	143	130	134	113	32	14	9.2	45
F-8	.01	8.9	8.1	8.3	7.4	143	128	131	113	32	14	6.3	30
F-9	.01	8.7	7.9	8.3	7.3	139	123	131	111	32	14		
F-10	.01	8.7	8.0	8.3	7.3	139	125	131	111	32	14	9.0	45
F-11	.02	8.8	8.0	8.3	7.3	141	125	131	111	32	14		
F-12	.01	8.8	8.0	8.3	7.3	141	125	131	111	32	14	10.5	45
F-13	.01	8.8	8.0	8.3	7.3	141	125	131	111	32	14		
F-14	.01	8.9	8.2	8.3	7.3	143	130	131	111	32	14	12.8	50
F-15	.02	9.0	8.2	8.4	7.3	145	130	133	111	32	14		
F-16	.02	9.0	8.2	8.5	7.4	145	130	135	113	32	14	7.9	35

TABLE XII: CALCULATED RESULTS

Run No.	Temp. °C	Rate (gms/hr)	Still Press. (Microns)	Vapor Press. (mm Hg)	Efficiency (per cent)
A-8	122	10.0	.01	0.5	.143
A-10	122	10.0	.01	0.5	.143
A-12	122	9.9	.01	0.5	.141
A-13	122	9.8	.01	0.5	.140
A-15	122	9.5	.01	0.5	.136
B-4	127	16.5	.01	0.7	.197
B-6	125	9.3	.01	0.6	.121
B-8	125	8.7	.01	0.6	.113
B-10	125	8.7	.01	0.6	.113
B-12	125	8.5	.01	0.6	.110
B-14	125	8.4	.01	0.6	.109
B-16	125	8.5	.01	0.6	.110
C-2	123	4.6	.02	0.6	.066
C-4	123	3.6	.03	0.6	.052
C-6	123	4.1	.03	0.6	.059
C-8	125	5.1	.03	0.6	.073
C-10	125	7.4	.02	0.6	.096
C-12	127	11.8	.02	0.7	.148
C-14	127	11.2	.02	0.7	.140
C-16	131	10.8	.01	0.8	.118
D-7	131	11.6	.01	0.8	.126
D-9	131	11.8	.01	0.8	.129
D-12	133	24.1	.01	0.8	.241
D-15	133	22.4	.01	0.8	.224
E-2	143	6.8	.09	1.2	.052
E-4	143	28.0	.03	1.2	.215
E-5	143	21.2	.03	1.2	.149
E-7	147	22.7	.03	1.4	.160
E-9	149	11.8	.09	1.5	.071
E-11	149	15.6	.01	1.5	.104
E-13	151	9.1	.09	1.6	.054
E-18	149	15.6	.01	1.5	.104
E-20	141	13.0	.02	1.1	.108
E-22	133	12.1	.02	0.8	.121
E-24	137	10.8	.01	1.0	.100
E-26	135	11.4	.02	0.9	.110
E-27	153	18.9	.01	1.6	.115
F-1	143	19.3	.01	1.2	.148
F-3	143	15.1	.01	1.2	.117
F-8	143	13.2	.01	1.2	.101
F-10	139	12.6	.02	1.0	.108
F-12	141	14.7	.01	1.1	.120
F-14	143	16.1	.01	1.2	.124
F-16	145	14.3	.02	1.3	.106

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APPENDIX III

(PHOTOGRAPHS)

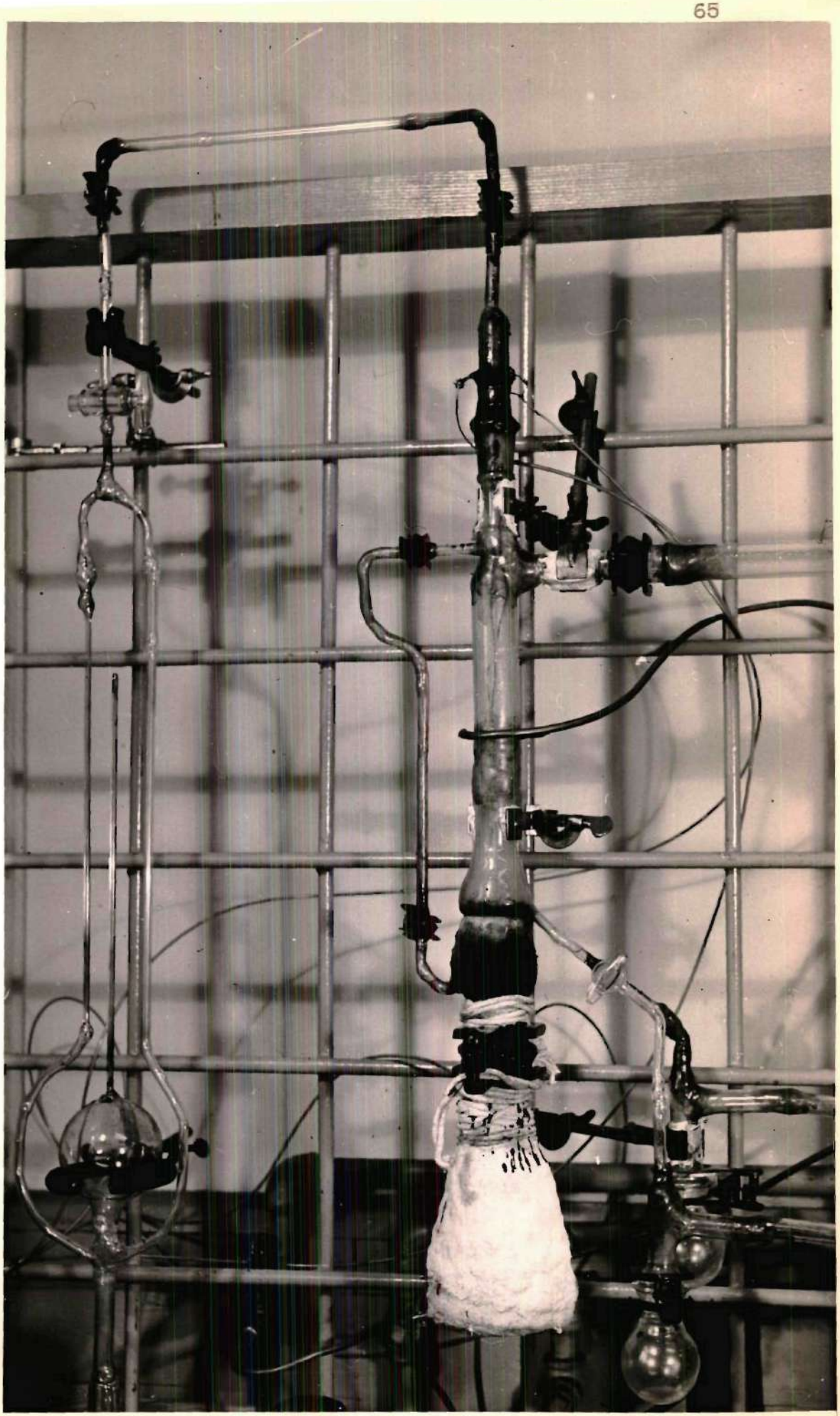

FIGURE 18: DISTILLATION SYSTEM

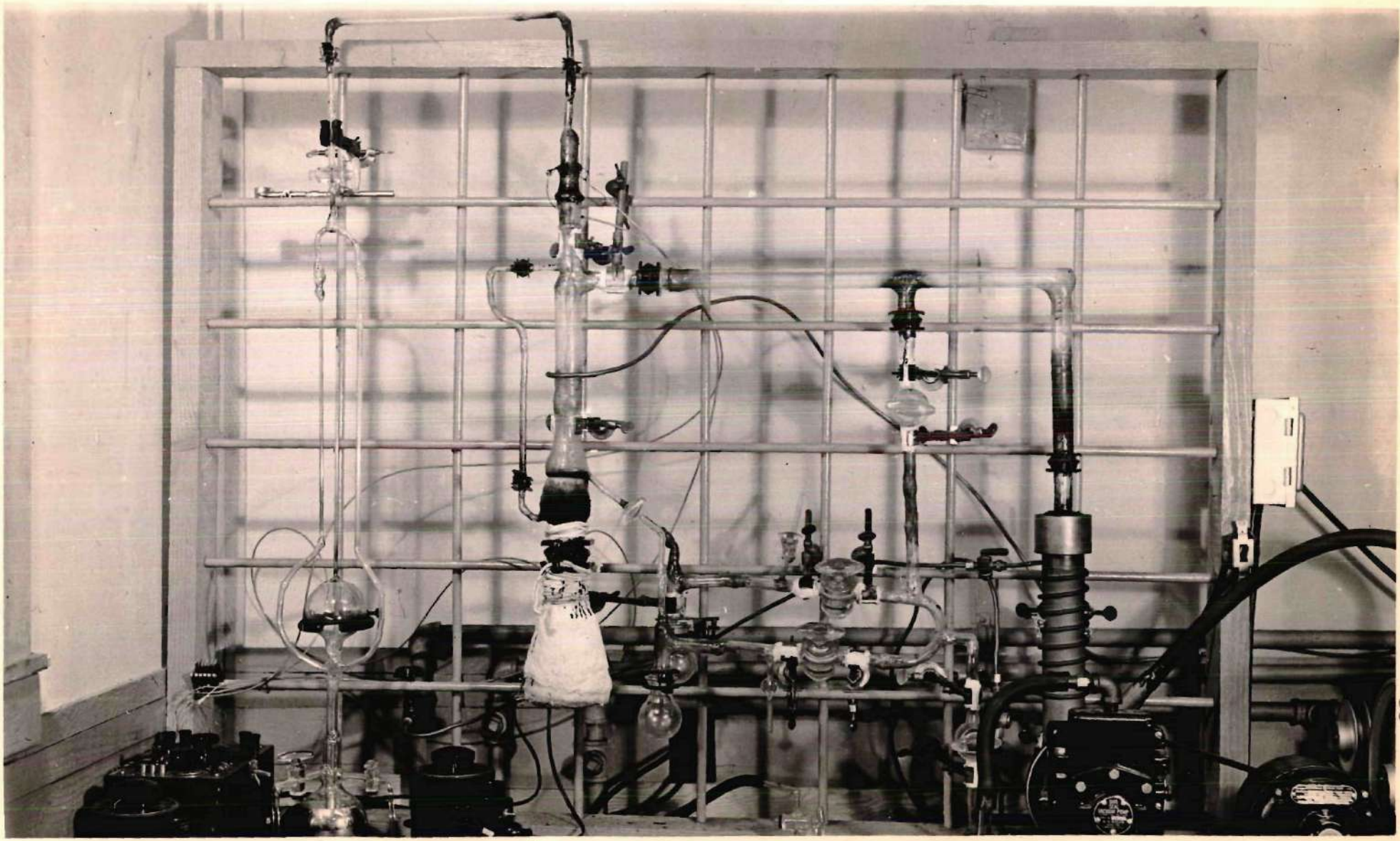

FIGURE 19: OVERALL VIEW OF EQUIPMENT